

Nanomaterials Science at the Advanced Photon Source

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NNI Workshop on X Rays and Neutrons:
Essential tools for nanoscience research

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X-ray tools for nanomaterials science

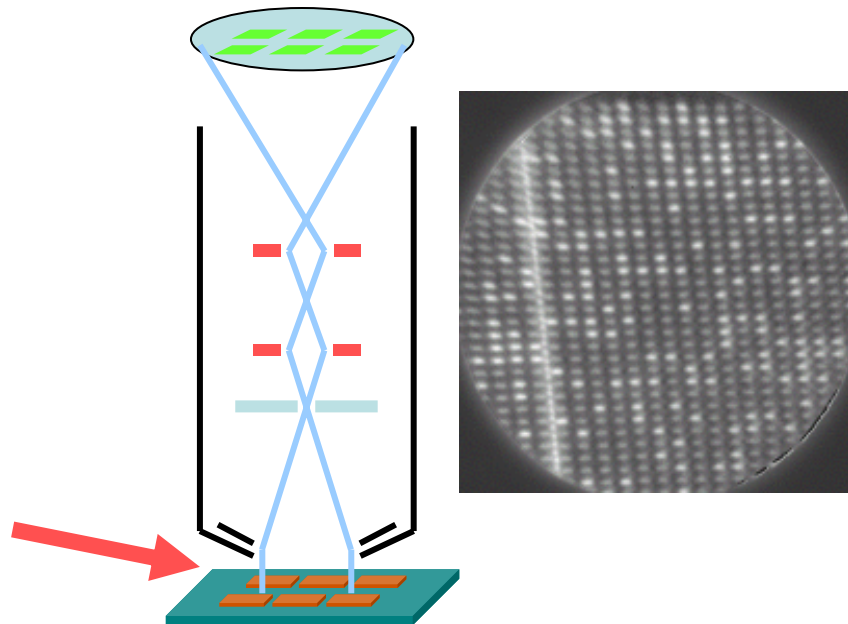
– emphasis on *in-situ* and real time

- The CNM hard x-ray nanoprobe
 - 20 nm probe
 - Full-field imaging
- Complementary **imaging facilities** at the APS
 - X-ray excited optical luminescence (XEOL)
 - Soft-x-ray photoemission electron microscopy (PEEM)
 - Scanning fluorescence and scanning nano-diffraction
- Complementary **x-ray scattering** facilities at the APS
 - X-ray reflectivity
 - SAXS and GISAXS
 - Coherent diffraction (“lensless” imaging)
 - Diffraction under *in-situ* growth
 - X-ray photon correlated spectroscopy (XPCS)

Polarization dependent imaging

Helicity-dependent X-ray emission provides information concerning spin polarized density of bulk occupied states

Photoemission Microscopy



Spatial resolution target of 20 nm

• **Magnetic contrast:**

- Domain imaging
- Ground states in nanoscale systems
- Interactions in particle arrays
- Finite size effects

• **Chemical contrast**

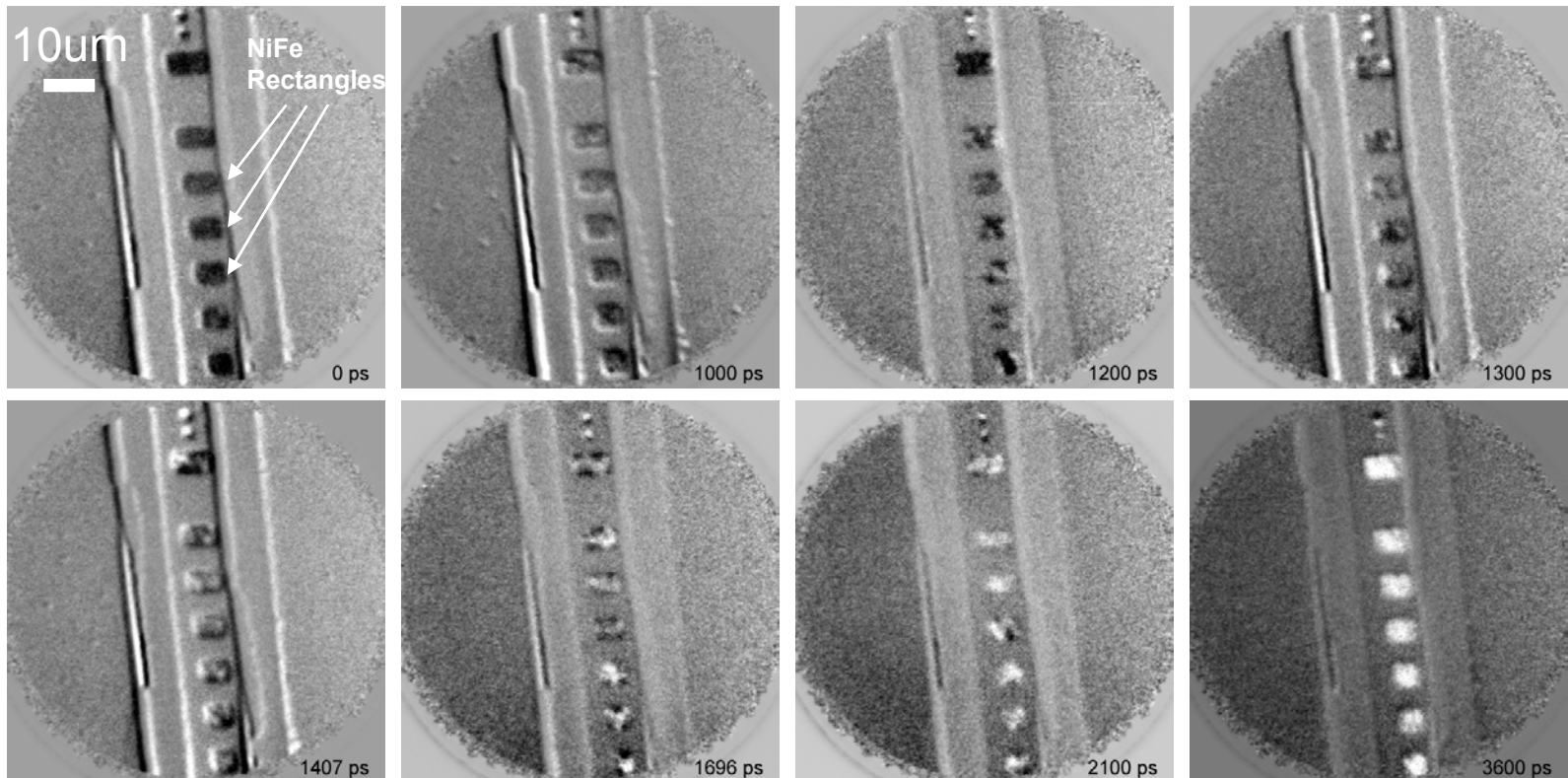
- Self-assembled systems
- Segregation
- Local electronic structure
- Buried layers (~5 nm)

• **Soft x-ray advantages:**

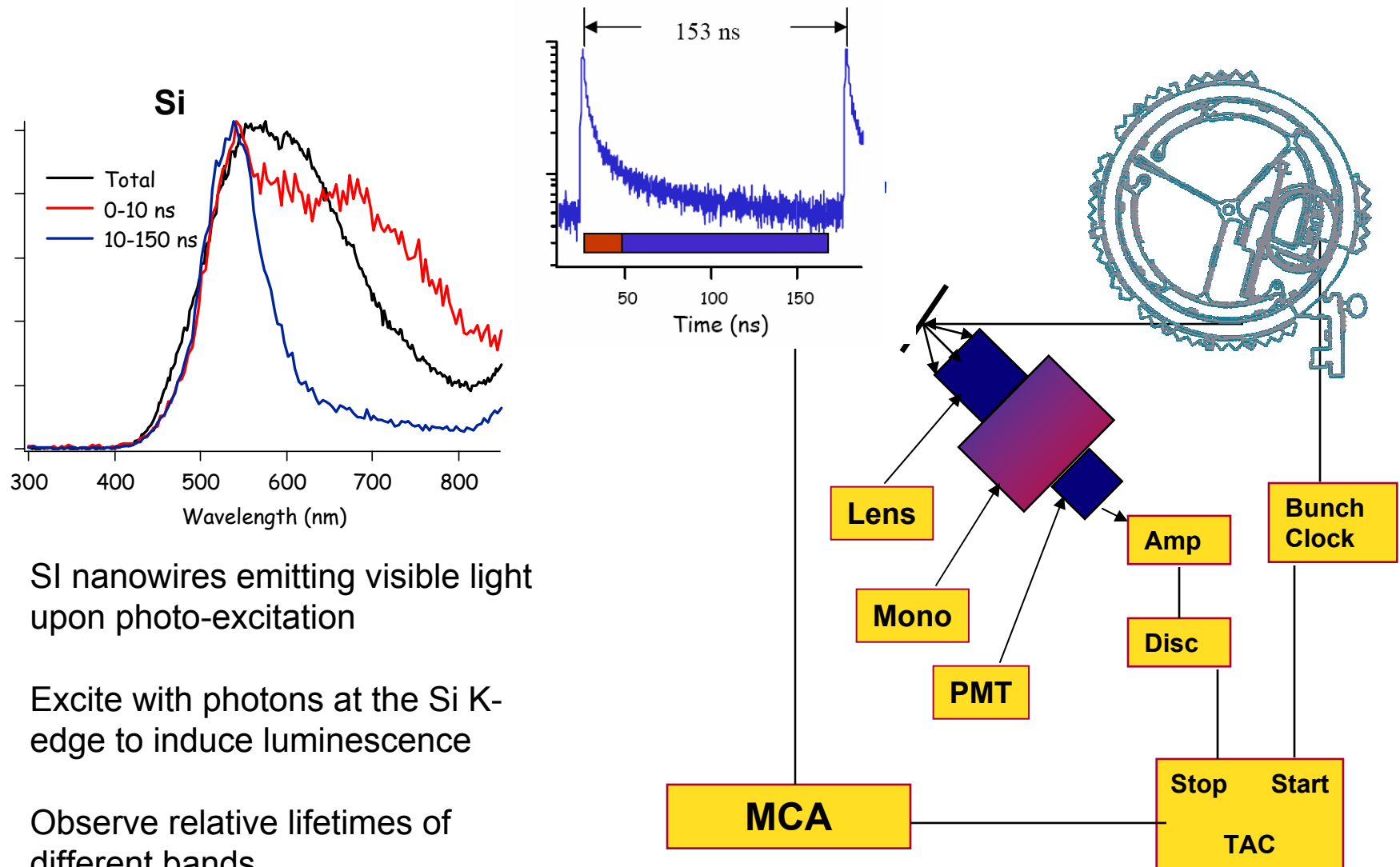
- High magnetic contrast
- Access to TM, RE, semiconductors

Time-resolved Photoemission electron microscopy (PEEM)

- NiFe dots with varying aspect ratios grown on top of a coplanar waveguide
- Apply a time-dependent field synchronized with the storage ring
- The dots respond to a positive to negative field step.
- Follow the domain pattern during the reversal.
- Full reversal of the magnetization in 3.6 ns.



Time-resolved X-ray-excited optical luminescence (XEOL)



Si nanowires emitting visible light upon photo-excitation

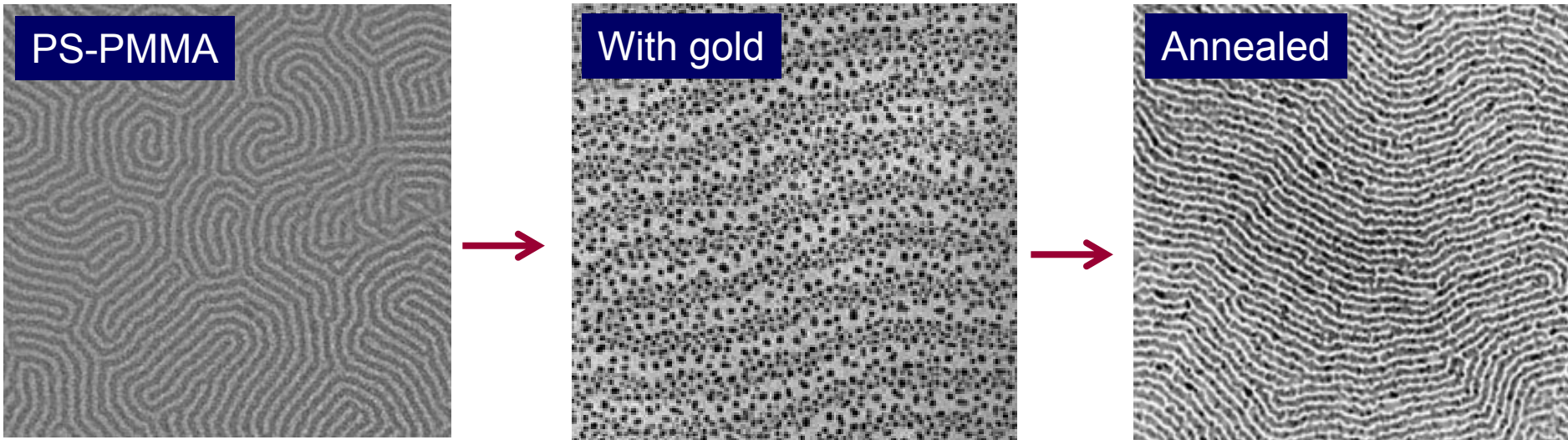
Excite with photons at the Si K-edge to induce luminescence

Observe relative lifetimes of different bands

Heirarchical self-assembly: not an equilibrated process

- Hierarchical self-assembly of metal nanostructures on an ultrathin diblock copolymer scaffold

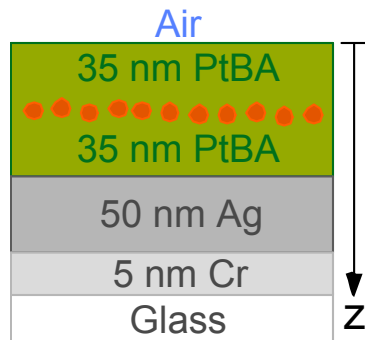
Lopes & Jaeger, Nature 414, 735 (2001)



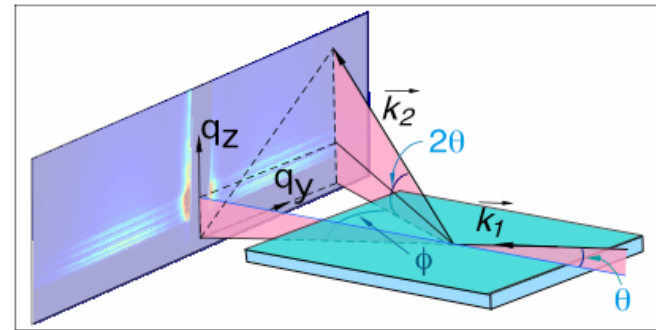
- Self-assembly of nanostructure is mostly an art.
- Controlled self-assembly needs to be guided by a thorough understanding of particle diffusion kinetics.
- Diffusion in thin films can be drastically different from that in bulk.
- Understanding the diffusion of nanoscale metal particles in ultrathin polymer films in a confined environments is critical.
- GISAXS using x-ray wave-guides: in-plane motion of nanoparticles

Diffusion of Nanoparticles in Ultrathin Films by GISAXS

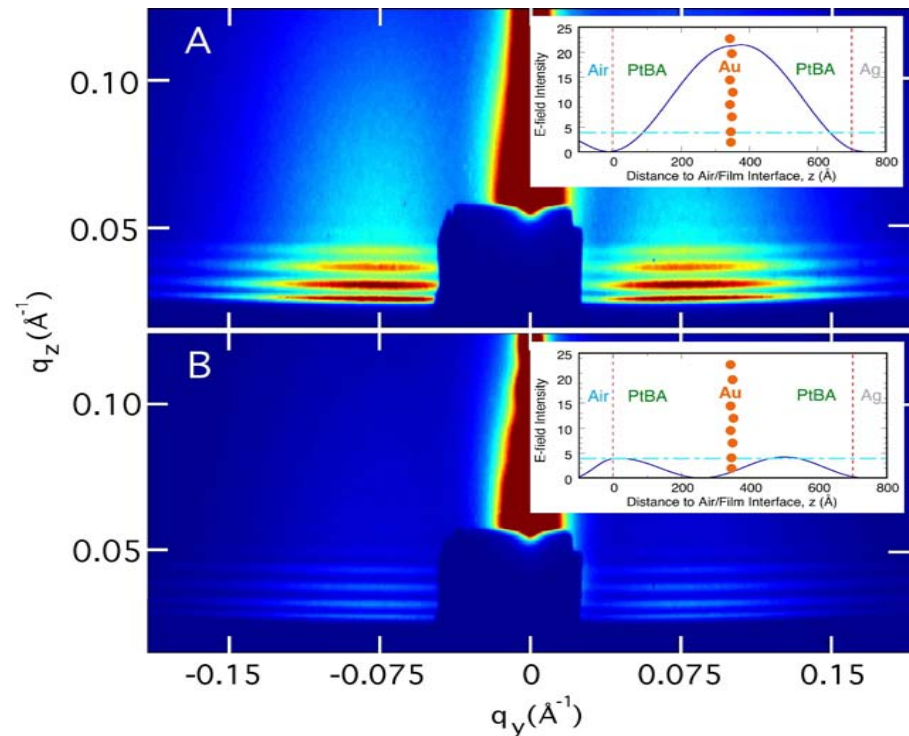
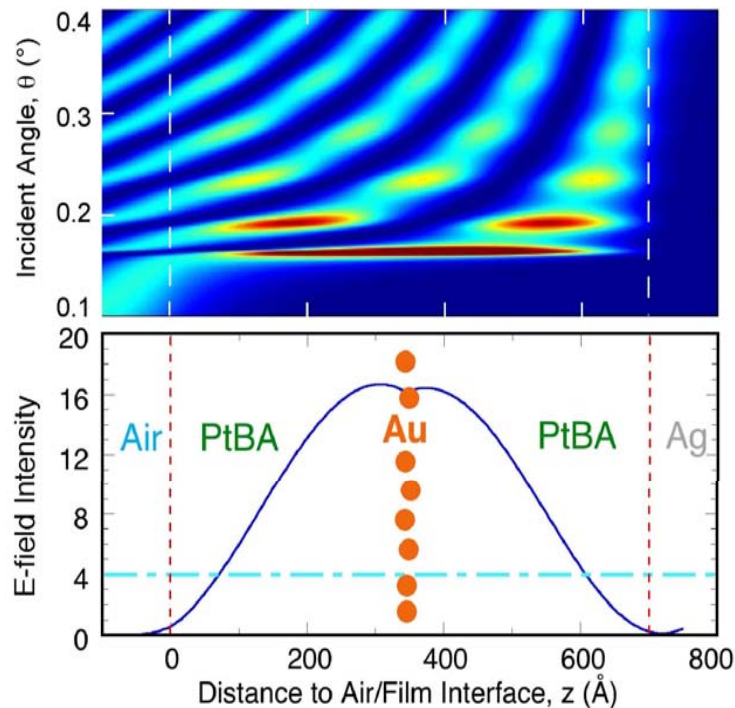
X-ray waveguiding: 1D nanoprobe perpendicular to interfaces



Waveguiding effect: Wang,
et al. Science **258**, 775
(1992)

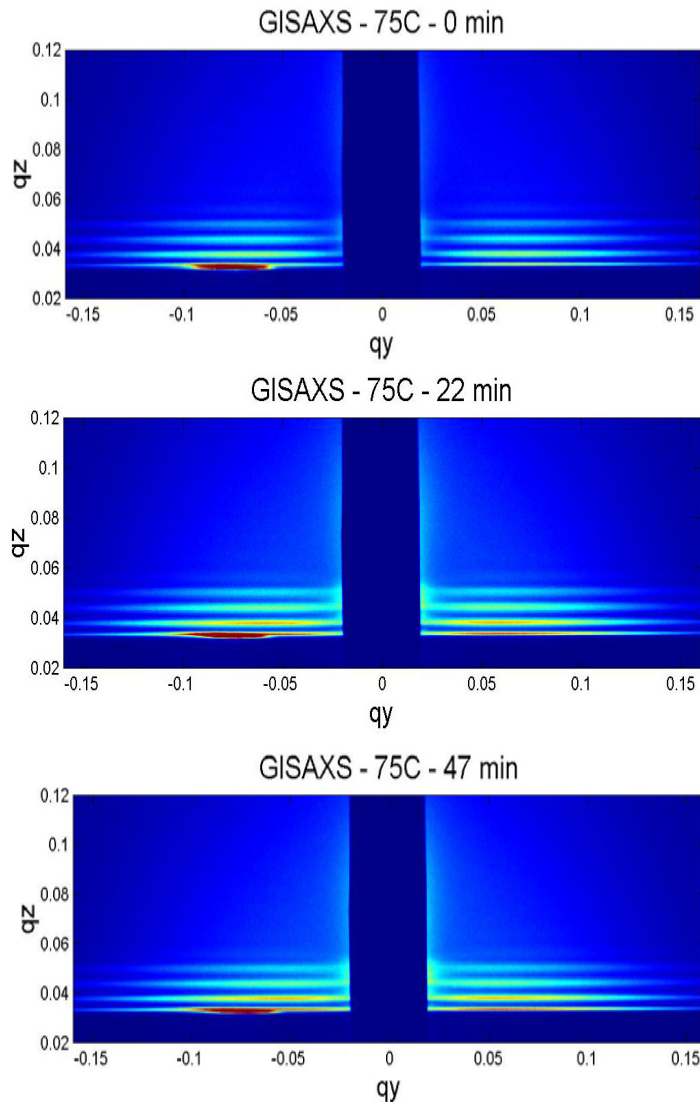


Electric Field distribution and enhancement

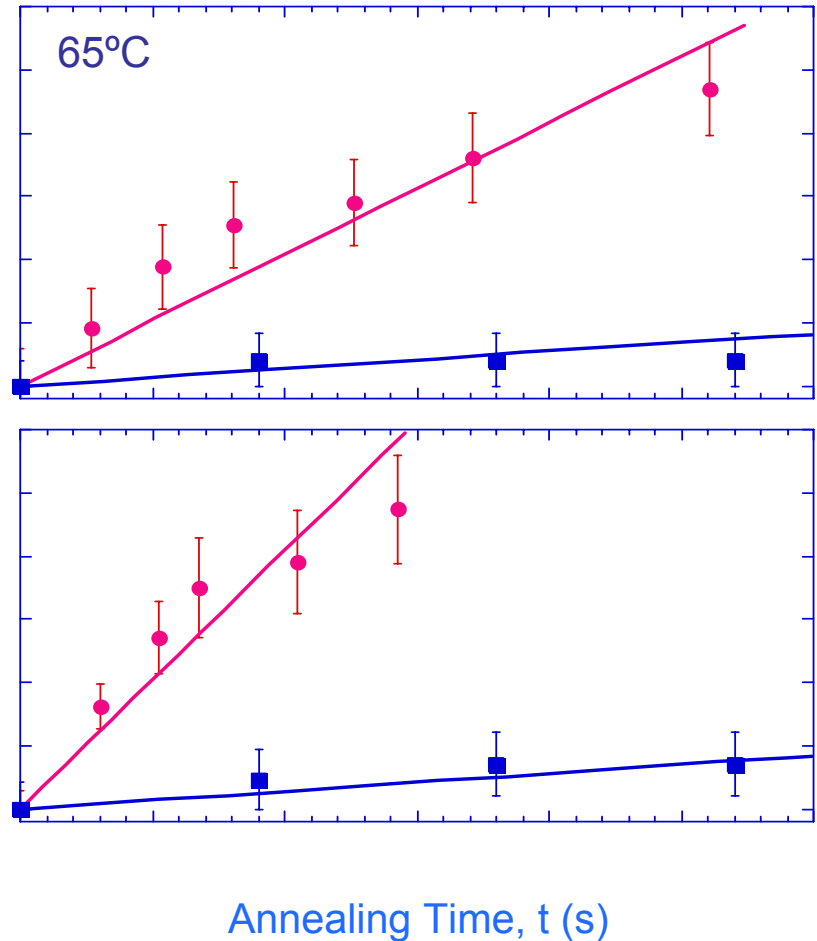


Diffusion of Nanoparticles in Ultrathin Films by GISAXS

Real time GISAXS reveals much faster in-plane motion



Mean Square Displacement (\AA^2)

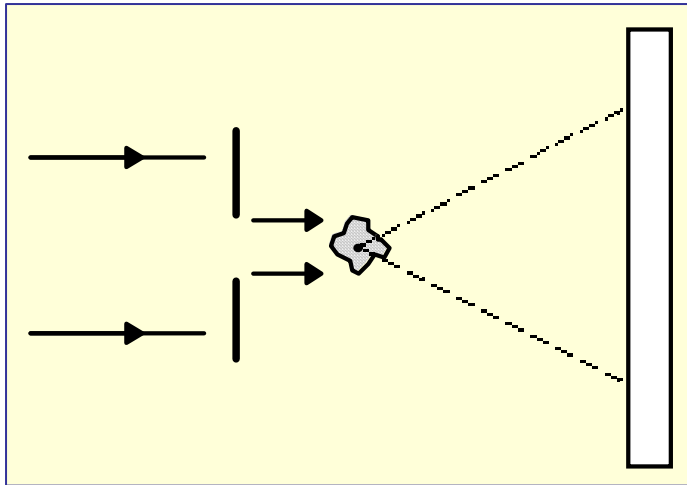


- **A much faster in-plane motion facilitates the formation of 2D nanostructure**

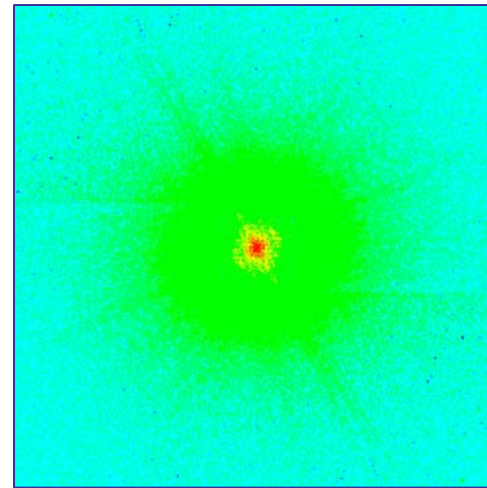
Guico, Narayanan, Wang, Shull, Macromolecules, **37**, 8357 (2004)

Narayanan, Lee, Guico, Sinha, Wang, Phys. Rev. Lett. **94**, 145504 (2005)

X-ray coherent diffraction



J. Miao, et al., *Nature* 400, 342 (1999)



Diffraction pattern from
E. coli bacteria (log scale)

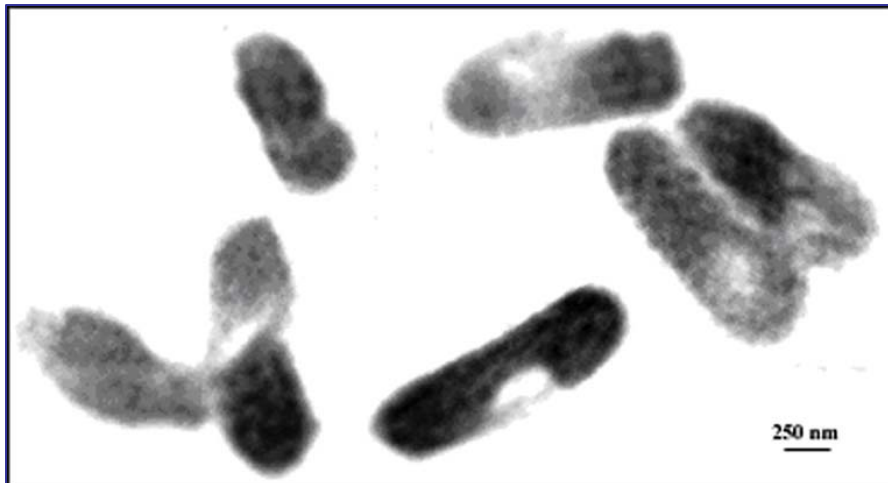


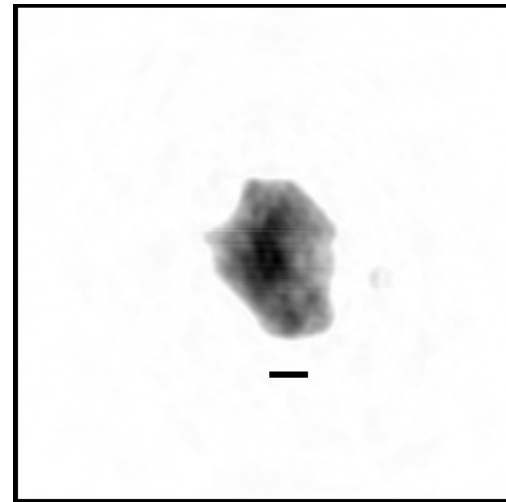
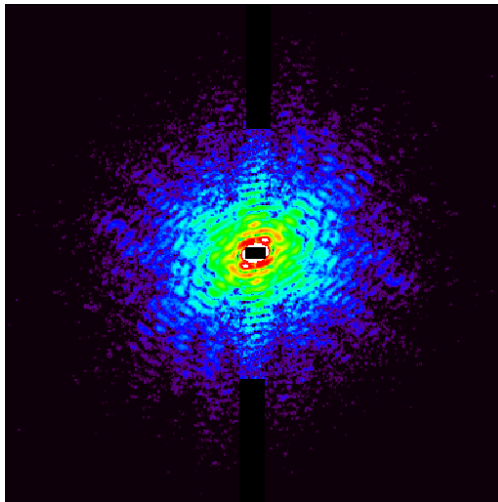
Image reconstructed
from diffraction
pattern to ~ 30 nm
resolution

J. Miao, et al., *Proc. Natl. Acad. Sci.* 100, 110 (2003)

Fishbone at different mineralization stages

Mineralized Bone (Fish bone particles at different mineralization stages):

1. Development and aggregation of calcium apatite nano-crystals in collagen protein matrix during mineralization
2. Structural aspect of the mineral phase in bone is poorly understood



Low degree mineralized fish bone and its reconstructed image.
The scale bar corresponds to 500 nm

Summary

- Need characterization methods that are effective in real time and in situ
- Showed a few examples
 - PEEM
 - XEOL
 - Resonant enhanced (waveguide) GISAXS